



Pyriproxyfen/129032/Valent USA Corporation
OPPTS 860.1500
Crop Field Trial - Mustard Greens

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Registration Action Branch 1
(RAB1)
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STUDY REPORT:

MRID No. 455560-09 C.A. Green (2001) Magnitude of the Residues of Pyriproxyfen on Mustard Greens. Laboratory Project Identification Number: 20191. Unpublished study prepared by Valent USA Corporation. 172 pages.

EXECUTIVE SUMMARY:

Supervised crop field trials were conducted in Wisconsin, Texas, California, Louisiana, South Carolina, and Mississippi in/on mustard greens treated two times at an application rate of 0.066 lb ai/A (seasonal rate of 0.132 lb a.i./A) with a pre-harvest interval (PHI) of 6-8 days and a retreatment interval (RTI) of 14 days. The results from these trials show that pyriproxyfen residues ranged from 0.23-1.69 ppm in treated mustard greens samples. Pyriproxyfen residues demonstrated a rapid decline with increasing PHI over a range of 3-14 days. Doubling the application rate resulted in a significant ($>2x$) increase in pyriproxyfen residue level. However, the PHI and application rate trends were based on the results of just one trial each. The limit of quantitation (LOQ) was established at 0.02 ppm.

COMPLIANCE:

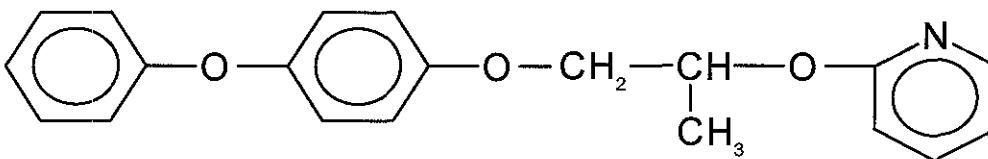
Signed and dated GLP, Quality Assurance and Data Confidentiality statements were provided. Six minor GLP deviations were noted, although they did not impact the validity of the study.



Pyriproxyfen/129032/Valent USA Corporation
OPPTS 860.1500
Crop Field Trial - Mustard Greens

A. BACKGROUND INFORMATION

Pyriproxyfen is an analogue of an insect juvenile hormone and interferes with the hormonal control of insect growth and development, thereby inhibiting egg hatch, larval embryogenesis, metamorphosis, and adult emergence. There are currently three end-use products of pyriproxyfen with food/feed uses that are registered to Valent: two emulsifiable concentrates (EC) and a wettable powder (WP). These formulations are registered for use on bushberry, citrus fruits, cotton, fruiting vegetables, guava, lychee, pome fruits, stone fruits, and tree nuts, and are marketed under the trade names KNACK[®] Insect Growth Regulator [0.86 lb/gal EC; EPA Reg. No. 59639-95], ESTEEM[®] Insect Growth Regulator [2.9 lb/gal EC; EPA Reg. No. 59639-104], and ESTEEM[®] 35 WP Insect Growth Regulator [35% WP; EPA Reg. No. 59639-115].

TABLE A.1. Test Compound Nomenclature	
Compound $C_{20}H_{19}NO_3$	Chemical Structure 
Common name	Pyriproxyfen
IUPAC name	4-phenoxyphenyl (RS)-2-(2-pyridyloxy)propyl ether
CAS name	2-[1-methyl-2-(4-phenoxyphenoxy)ethoxy]pyridine
CAS #	95737-68-1
End-use product/EP	Knack IGR, Esteem IGR



Pyriproxyfen/129032/Valent USA Corporation
OPPTS 860.1500
Crop Field Trial - Mustard Greens

B. EXPERIMENTAL DESIGN

B.1. Study Site Information

No unusual weather events were noted at any of the field trial sites involved in the present study.

TABLE B.1.1. Study Use Pattern.								
Location (City, State/Year)	EP ¹	Application						Tank Mix Adjuvants
		Timing	Rate, lb a.i./A	RTI ² (days)	Treat. No.	Method	Total Rate, lb a.i./A	
Pepin County, WI/1999	Knack IGR (0.86 EC)	1) 21 (\pm 1) days before harvest 2) 7 (\pm 1) days before harvest	1) 0.068 2) 0.068	14	2	tractor-mounted boom sprayers	0.136	None
Hidalgo County, TX/1999	Knack IGR (0.86 EC)	1) 21 (\pm 1) days before harvest 2) 7 (\pm 1) days before harvest	1) 0.066 2) 0.064	14	2	tractor-mounted boom sprayers	0.130 (1x)	None
			1) 0.136 2) 0.135				0.271 (2x)	
Santa Cruz County, CA/1999	Knack IGR (0.86 EC)	1) 21 (\pm 1) days before harvest 2) 7 (\pm 1) days before harvest	1) 0.066 2) 0.065	14	2	tractor-mounted boom sprayers	0.130	None
St. Landry Parish, LA/2000	Knack IGR (0.86 EC)	1) 21 (\pm 1) days before harvest 2) 7 (\pm 1) days before harvest	1) 0.067 2) 0.067	14	2	tractor-mounted boom sprayers	0.134	None
Barnwell County, SC/2000	Knack IGR (0.86 EC)	1) 21 (\pm 1) days before harvest 2) 7 (\pm 1) days before harvest	1) 0.067 2) 0.068	14	2	tractor-mounted boom sprayers	0.135	None
Washingto n County, MS/2000	Knack IGR (0.86 EC)	1) 21 (\pm 1) days before harvest 2) 7 (\pm 1) days before harvest	1) 0.066 2) 0.066	14	2	tractor-mounted boom sprayers	0.133	None

¹EP = End-use Product

²Retreatment Interval



Pyriproxyfen/129032/Valent USA Corporation
OPPTS 860.1500
Crop Field Trial - Mustard Greens

TABLE B.1.3. Trial Numbers and Geographical Locations															
Crop		NAFTA Growing Regions													Total trials
		1	2	3	4	5	6	7	8	9	10	11	12	13	
Mustard greens	Submitted		1		2	1	1				1				6
	Requested		1		1	1	1				1				5

B.2. Analytical Methodology

The samples were analyzed using minor modifications (equivalent glassware and slightly different GC parameters) to the reference method: "Determination of Pyriproxyfen Residues in Apples, Pears, and Citrus Fruits", Valent method RM-33P-1-3a. In brief, residues of pyriproxyfen were extracted from samples with acetone, partitioned with dichloromethane/water, and cleaned using silica gel column chromatography. Quantitation for pyriproxyfen was by gas chromatography with a nitrogen-phosphorus detector (GC/NPD). The limit of quantitation (LOD) was established at 0.02 ppm; the limit of detection (LOD) estimate was 0.01 ppm.

C. RESULTS AND DISCUSSION

The number of trials and the geographic representation are both adequate for mustard greens as a representative commodity of crop group 5, where five field trials are requested (OPPTS 860.1500, Table 2). When treated at a seasonal application rate of 0.132 lb ai/A with a PHI of 6-8 days and a 14-day RTI, pyriproxyfen residues ranged from 0.23-1.69 ppm in the treated mustard greens samples analyzed. The residue decline data submitted indicates that pyriproxyfen residues decrease rapidly when the PHI is increased from 3-14 days.

Concurrent recovery results indicate that the data collection method is adequate for detecting pyriproxyfen residues in/on mustard greens samples (see Table C.1). Satisfactory recovery values were obtained from mustard greens samples spiked at 0.02 and 0.10 ppm. All untreated control samples were free of pyriproxyfen residues and interferences.

No storage stability study was conducted as the longest harvest-to-analysis interval of any field treated sample was less than 30 days.

As shown in Tables C.2 and C.3, pyriproxyfen residue levels in mustard greens samples ranged from 0.023-1.69 ppm when the PHI was 6-8 days, and the RTI was 14 days.

TABLE C.1. Summary of Concurrent Recoveries of Pyriproxyfen from Mustard greens.



Pyriproxyfen/129032/Valent USA Corporation
OPPTS 860.1500
Crop Field Trial - Mustard Greens

Matrix	Analyte	Spike level (mg/kg)	Sample size (n)	Recoveries (%)	Mean \pm std dev
Mustard greens	Pyriproxyfen	0.020	8	74-110	92 \pm 13
		0.10	8	82-105	95 \pm 7

TABLE C.2. Residue Data from Crop Field Trials with Pyriproxyfen.

Location (City, State/Year)	Region	Crop/ Variety	Application Rate, lbs ai/A	Total Rate, lbs ai/A	RTI (days)	PHI (days)	Residues (ppm)
Pepin County, WI/1999	5	Mustard Green/ Florida Broadleaf	0.0681 0.0677	0.136	14	3	1.6, 2.0
						7	0.30, 0.39
						10	0.34, 0.31
						14	0.05, 0.04
Hidalgo County, TX/1999	6	Mustard Green/ Savannah	0.0655 0.0644	0.130	14	7	0.29, 0.38
			0.136 0.135	0.271	14	7	1.4, 1.2
Santa Cruz County, CA/1999	10	Mustard Green/ Southern Giant Curled	0.0655 0.0646	0.130	14	7	0.35, 0.23
St. Landry Parish, LA/2000	4	Mustard Green/ Florida Broadleaf	0.0666 0.0672	0.134	14	6	1.10, 1.21
Barnwell County, SC/2000	2	Mustard Green/ Florida Broadleaf	0.0672 0.0679	0.135	14	7	1.69, 1.53
Washington County, MS/2000	4	Mustard Green/ Florida Broadleaf	0.0664 0.0661	0.133	14	8	0.49, 0.43

TABLE C.3. Summary of Residue Data from Crop Field Trials with Pyriproxyfen.

Commodity	Total Applic. Rate, lb a.i./A	PHI (days)	Analyte	Residue Levels (ppm)				
				Min.	Max.	HAFT*	Mean	Std. Dev.
Mustard Greens	0.132	6-8	Pyriproxyfen	0.29	1.69	1.61	0.70	0.53

* HAFT = Highest Average Field Trial.

D. CONCLUSION



Pyriproxyfen/129032/Valent USA Corporation
OPPTS 860.1500
Crop Field Trial - Mustard Greens

The crop field trials for mustard greens are classified as acceptable and satisfy the guideline requirements for crop field trials (Residue Chemistry Guidelines OPPTS 860.1500) as they apply to "Vegetable, brassica, leafy, group 5."

E. STUDY DEFICIENCIES/CLARIFICATIONS

None.